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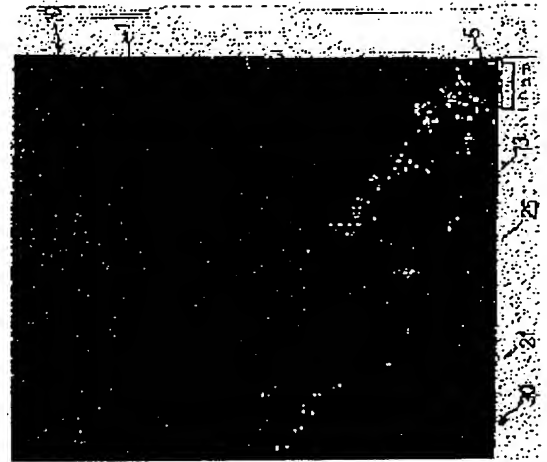
(72)Inventor : FUJIMORI NOBUHIKO
KAWADA TSUNEHIRO

(54) R-T-B SINTERED MAGNET, RING MAGNET AND VOICE COIL MOTOR

(57)Abstract:

PROBLEM TO BE SOLVED: To provide an R-T-B sintered magnet which is manufactured by the use of R-T-B sintered magnet alloy powder through a reduction diffusion method, inexpensive, and high in performance, a ring magnet and a voice coil motor.

SOLUTION: An R-T-B sintered magnet contains an R2T14B intermetallic compound (R denotes one or more rare earth elements, including Y; and T is Fe or Fe and Co) as a main phase, is below 0.02 wt.% (not zero) in avoidable Ca content, and provided with a main phase crystal grain part which resides through a rare earth rich phase, containing at least two or more parts that are a core part and a surface both formed of main phase crystal grains, where the main phase crystal grains of the surface are 0.5 to 50 nm in average crystal grain diameter.



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(71) 出願人 000005083

日立金属株式会社

東京都港区芝浦一丁目2番1号

(72) 発明者 藤森 信彦

埼玉県熊谷市三ヶ尻5200番地日立金属株式会社磁性材料研究所内

(72) 発明者 川田 常宏

埼玉県熊谷市三ヶ尻5200番地日立金属株式会社磁性材料研究所内

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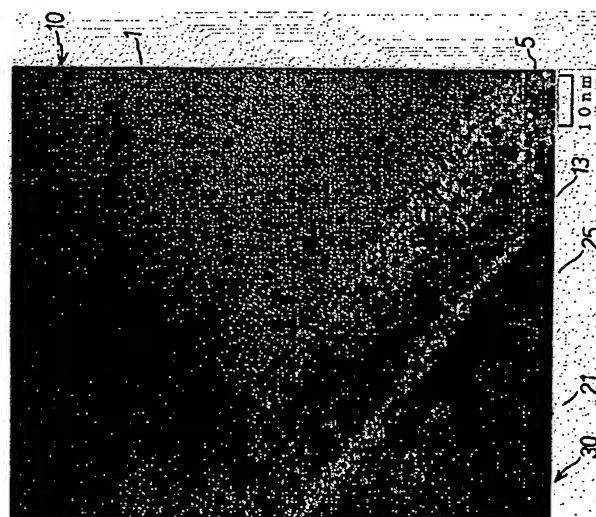
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(54) 【発明の名称】 R-T-B系焼結磁石、リング磁石およびボイスコイルモータ

(57) 【要約】

【課題】 還元拡散法によるR-T-B系焼結磁石用合金粉末を用いて製造された安価でかつ高性能のR-T-B系焼結磁石、リング磁石およびボイスコイルモータを提供する。

【解決手段】 $R_2T_{14}B$ 型金属間化合物 (RはYを含む希土類元素の1種または2種以上であり、TはFeまたはFeとCoである) を主相とするR-T-B系焼結磁石であって、不可避に含有されるCa量が0.02重量%未満 (0を含まず) であり、希土類リッチ相を介して存在する主相結晶粒部分が芯部および表層部の少なくとも2個以上の主相結晶粒からなるものを含むとともに前記表層部の主相結晶粒の平均結晶粒径が0.5~50nmであるR-T-B系焼結磁石。



【特許請求の範囲】

【請求項1】 $R_2T_{14}B$ 型金属間化合物（ R は Y を含む希土類元素の1種または2種以上であり、 T は Fe または Fe と Co である）を主相とする $R-T-B$ 系焼結磁石であって、

不可避に含有される Ca 量が0.02重量%未満（0を含まず）であり、希土類リッチ相を介して存在する主相結晶粒部分が芯部および表層部の少なくとも2個以上の主相結晶粒からなるものを含むとともに前記表層部の主相結晶粒の平均結晶粒径が0.5~50nmであることを特徴とする $R-T-B$ 系焼結磁石。

【請求項2】 希土類リッチ相を介して存在する主相結晶粒部分を1個とカウントしたとき、前記の芯部および表層部からなる主相結晶粒部分の占める個数比率が100個の主相結晶粒部分あたり50%未満である請求項1に記載の希土類焼結磁石。

【請求項3】 重量%で、主要成分が R :27~33%、 B :0.8~1.5%、 M :0.01~1%（ M は Al 、 Ga 、 Nb 、 Cu の1種または2種以上）、残部 T からなり、不可避に含有される酸素量が0.6%未満、炭素量が0.2%以下および密度が7.53g/cm³以上であって、20℃における角型比(H_k/iH_c)が95%以上、最大エネルギー積(BH)maxが302.5kJ/m³ (38MG0e)以上である請求項1または2に記載の $R-T-B$ 系焼結磁石。

【請求項4】 $R_2T_{14}B$ 型金属間化合物（ R は Y を含む希土類元素の1種または2種以上であり、 T は Fe または Fe と Co である）を主相とする $R-T-B$ 系焼結磁石からなるリング磁石であって、不可避に含有される Ca 量が0.02重量%未満（0を含まず）であり、希土類リッチ相を介して存在する主相結晶粒部分が芯部および表層部の少なくとも2個以上の主相結晶粒からなるものを含むとともに前記表層部の主相結晶粒の平均結晶粒径が0.5~50nmであることを特徴とするリング磁石。

【請求項5】 $R_2T_{14}B$ 型金属間化合物（ R は Y を含む希土類元素の1種または2種以上であり、 T は Fe または Fe と Co である）を主相とする $R-T-B$ 系焼結磁石を用いたボイスコイルモータであって、前記焼結磁石は不可避に含有される Ca 量が0.02重量%未満（0を含まず）であるとともに希土類リッチ相を介して存在する主相結晶粒部分が芯部および表層部の少なくとも2個以上の主相結晶粒からなるものを含み、かつ前記表層部の主相結晶粒の平均結晶粒径が0.5~50nmであることを特徴とするボイスコイルモータ。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、還元拡散法による $R-T-B$ 系焼結磁石用合金粉末（ R は Y を含む希土類元素のうちの1種または2種以上であり、 T は Fe または Fe と Co である）を用いて製造された安価でかつ高

性能の $R-T-B$ 系希土類焼結磁石、リング磁石およびボイスコイルモータに関する。

【0002】

【従来の技術】 Nd 、 Dy または Pr 等の資源的に比較的豊富な希土類元素、 Fe または Fe と Co および B を主要成分とする $R-T-B$ 系焼結磁石は、従来にない高い磁気特性を有することから年々需要が増え、現状では希土類磁石市場の大半を占めるようになった。この需要増に伴い、 $R-T-B$ 系焼結磁石の原価低減が進み、より安価で高性能のものが要求されてきている。

【0003】 $R-T-B$ 系焼結磁石は $R-T-B$ 系焼結磁石組成に調整した合金粉末を圧縮成形、焼結、熱処理および表面処理して製造される。 $R-T-B$ 系合金粉末として主に $R-T-B$ 系溶製合金（ストリップキャスト合金または高周波溶解／ casting合金等）の粉碎粉末が用いられている。他方、安価な $R-T-B$ 系合金粉末として、希土類酸化物、 $Fe-B$ （ $Fe-Co-B$ ）合金粉末、 Fe 粉末および還元剤である金属 Ca を適量比率で配合し混合後、不活性ガス雰囲気中で加熱することにより、 Ca 還元／拡散反応を行い、その後室温まで冷却する。続いて、洗浄および真空（加熱）乾燥する工程：還元／拡散法（Reduction/Diffusion法）により作製される合金粉末（以下、これを R/D 粉末という）がある。

【0004】しかし、溶製合金粉末を用いて作製した $R-T-B$ 系焼結磁石に比べて、 R/D 粉末を用いて作製した $R-T-B$ 系焼結磁石は角型比および保磁力 iH_c 等の磁気特性が低いという問題がある。これは不可避に CaO 、酸素および炭素等を多量に含むためと判断される。前記不可避不純物を低減するために、 Ca 還元／拡散反応後の反応生成塊を洗浄液（水等）中に投入し、 CaO 等の反応副生成物を洗浄液中に効率よく抽出し洗い流す種々の工夫が施されている。また、 R/D 反応主生成物中に介在する反応副生成物を洗浄液中に抽出し易くするために機械的な粉碎を加えたり、あるいは洗浄時間を延長すると、洗浄中に酸化が進行したり、あるいは乾燥時の真空または不活性ガス雰囲気から大気に曝露した時点で酸化が顕著に進行するという問題がある。

【0005】上記問題の対策として、特開昭63-310905号公報では、低酸素、低 Ca の $R-T-B$ 系 R/D 粉末を得る製造方法として、 Ca 還元／拡散反応後の反応生成物の水洗においてインヒビター（腐食抑制剤）を $10^{-3} \sim 10^{-2}$ g/l添加した水を用いて水洗、脱水および真空乾燥する方法を開示している。しかしこの製造方法を採用した場合でも、工業生産上 $R-T-B$ 系 R/D 粉末の Ca 含有量はせいぜい0.05~0.1重量%程度までの低減が限界であることが本発明者らの検討からわかった。このため、前記 R/D 粉末のみを用いて $R-T-B$ 系焼結磁石を作製した場合、 $R-T-B$ 系溶製合金粉末（ Ca 含有量：ほぼ0ppm）を用いて作製した $R-T-B$ 系焼結磁石と比較すると Ca 含有量は非常に高くな

る。同時に、角型比 (Hk/iHc) が劣化して耐熱性の用途に適用できないという問題がある。Hkは $4\pi I-H$ 減磁曲線において、 $4\pi I$ (磁化の強さ) が0.9Brになる位置のH (磁界の強さ) 軸の読値である。Brは残留磁束密度である。前記問題は従来のR-T-B系統結磁石用のR/D合金粉末を所定量 (例えば10~100重量%) 配合して作製したR-T-B系統結磁石に共通する。

【0006】

【発明が解決しようとする課題】したがって、本発明の課題は、還元拡散法によるR-T-B系統結磁石用合金粉末 (RはYを含む希土類元素のうちの1種または2種以上であり、TはFeまたはFeとCoである) を用いて製造された安価でかつ高性能のR-T-B系統結磁石、リング磁石およびボイスコイルモータを提供することである。

【0007】

【課題を解決するための手段】上記課題を解決した本発明は、 $R_2T_{14}B$ 型金属間化合物 (RはYを含む希土類元素の1種または2種以上であり、TはFeまたはFeとCoである) を主相とするR-T-B系統結磁石であって、不可避に含有されるCa量が0.02重量%未満 (Oを含まず) であり、希土類リッチ相を介して存在する主相結晶粒部分が芯部および表層部の少なくとも2個以上の主相結晶粒からなるものを含むとともに前記表層部の主相結晶粒の平均結晶粒径が0.5~50nmであるR-T-B系統結磁石である。前記Ca含有量を有するとともに主相結晶粒部分が芯部および表層部の少なくとも2個以上の主相結晶粒からなる場合に、上記従来の還元拡散法によるR-T-B系統結磁石用合金粉末を用いた場合に比べて角型比 (Hk/iHc)、保磁力iHc等を顕著に向上することができる。また、前記焼結磁石において、希土類リッチ相を介して存在する主相結晶粒部分を1個とカウントしたとき、前記の芯部および表層部からなる主相結晶粒部分の占める個数比率が100個の主相結晶粒部分において50%未満である場合に、上記従来の還元拡散法によるR-T-B系統結磁石用合金粉末を用いた場合に比べて角型比 (Hk/iHc)、保磁力iHc等を顕著に向上したR-T-B系統結磁石が得られる。また、前記焼結磁石は、重量%で、主要成分組成がR:27~33%、B:0.8~1.5%、M:0.01~1% (MはAl、Ga、Nb、Cuの1種または2種以上)、残部Tからなり、不可避に含有される酸素量が0.6%未満、炭素量が0.2%以下および密度が7.53g/cm³以上のときに、20℃における角型比 (Hk/iHc) が95%以上、最大エネルギー積 (BH)_{max}が302.5 kJ/m³ (38MGOe) 以上になり、好ましい。

【0008】本発明の焼結磁石の製造に際し、成形体に行う真空加熱条件について以下に説明する。まず、還元拡散法により製造されたR-T-B系統結磁石用合金粉末を所定粒径に粉碎後、磁場中成形して成形体を得る。次に、成形体を $133\sim 1.2\times 10^{-3}$ Pa (1×10^0

$\sim 9\times 10^{-6}$ Torr) の真空中で850~1050℃に加熱する脱Caのための熱処理を行う。特に、前記成形体に $133\sim 1.2\times 10^{-3}$ Pa ($1\times 10^0\sim 9\times 10^{-6}$ Torr) の真空中で550~650℃に加熱する1段目の真空加熱処理と、続いて $133\sim 1.2\times 10^{-3}$ Pa ($1\times 10^0\sim 9\times 10^{-6}$ Torr) の真空中で850~1050℃に加熱する2段目の真空加熱処理とを行うことにより、脱Caが顕著になるとともに酸素量が低減されるので好ましい。真空加熱処理条件は、1段目の真空加熱処理の場合、 $133\sim 1.2\times 10^{-3}$ Pa ($1\times 10^0\sim 9\times 10^{-6}$ Torr) の真空度でかつ850~1050℃の加熱温度を選択することが脱Caのために好ましい。この真空加熱処理で除去されるCaは金属Caの状態で存在する分と考えられる。すなわち、1段目の真空加熱処理の加熱温度は金属Caの融点 (851℃) 以上がよく、かつ焼結温度より低い1050℃以下が好ましい。1050℃超では焼結による緻密化が急激に進んで脱Caが困難なためである。さらに好ましい1段目の真空加熱処理温度の範囲は900~1000℃である。1段目の真空加熱処理の加熱時間は0.5~5時間が好ましい。0.5時間未満では脱Caが十分に行われず、5時間超では脱Ca反応が飽和しかつ熱処理コストが増大する。真空度は工業生産上 $133\sim 1.2\times 10^{-3}$ Pa ($1\times 10^0\sim 9\times 10^{-6}$ Torr) であれば脱Caを実現するのに十分である。 133 Pa (1×10^0 Torr) 未満では脱Caが困難であり、 1.2×10^{-3} Pa (9×10^{-6} Torr) 超の高真空の採用は実用性に乏しい。2段目の真空加熱処理の場合、1段目の真空加熱処理条件として $133\sim 1.2\times 10^{-3}$ Pa ($1\times 10^0\sim 9\times 10^{-6}$ Torr) の真空中でかつ550~650℃の加熱条件を採用することが酸素量の低減の点から特に好ましい。これは、反応副生成物であるCa(OH)₂がCaOとH₂Oに分離する温度が580℃付近であることと相関があると判断される。本発明者らの検討から、約580℃で脱水化するように1段目の真空加熱処理の条件を設定すると脱Ca時の酸化が抑えられて酸素量を低減できることがわかった。550℃未満では脱水化が不十分であり、650℃超では反応副生成物の熱分解に起因する酸化が顕著になる傾向が認められた。1段目の真空加熱処理の加熱保持は0.5~5時間がよい。2段目の真空加熱処理条件の限定理由は上記と同様である。

【0009】本発明のR-T-B系統結磁石の組成限定理由を以下に説明する。以下、単に%と記しているのは重量%である。R量は27~33%が好ましい。R量が27%未満では実用に耐えるiHcの実現が困難であり、33%超ではBrが大きく低下する。実用上RとしてNd、Dy、Prの少なくとも1種を含むように選択される。B量は0.8~1.5%が好ましく、0.9~1.2%がより好ましい。B量が0.8%未満では実用に耐えるiHcの実現が困難であり、1.5%超ではBrが大きく低下する。Nb量は0.01~1%が好ましい。Nb量が0.01%未満では焼結時の結晶粒粗大化に対する抑制効果が得られず、1%超ではBrが

大きく低下する。A l 量は0.01~1%が好ましい。A l 量が0.01%未満ではiHcの向上効果が得られず、1%超ではBrが大きく低下する。G a 量は0.01~1%が好ましい。G a 量が0.01%未満ではiHcの向上効果が得られず、1%超ではBrが大きく低下する。C u 量は0.01~1%が好ましい。C u の微量添加はiHcの向上をもたらすが、0.01%未満では効果が認められず、1%超ではiHcの向上効果が飽和する。C o 量は0.3~5%が好ましい。C o 量が0.3%未満ではキュリー点および耐蝕性の向上効果が得られず、5%超ではBr、iHcが大きく低下する。回転機またはボイスコイルモータ等の耐熱用途に耐えるR-T-B系統結磁石、リング磁石を実現するために、不可避に含有される酸素量は0.6%未満が好ましく、0.4%以下がより好ましく、0.2%未満が特に好ましい。さらに、酸素量を0.3%以下に低減したとき、R含有量は、好ましくは28~32%、より好ましくは29~31%とすることがよい。炭素量の増加により希土類炭化物が増加して磁気特性が低下するので、炭素量は、好ましくは0.2%以下、より好ましくは0.1%以下がよい。

【0010】

【発明の実施の形態】本発明に係わる真空熱処理条件以外の好ましい製造条件について説明する。主要成分がR(Nd, Dy):30%、B:1.0%、Al:0.03%、残部Feとなるように、純度99.9%以上の(Nd, Dy)酸化物粉末、Fe-B粉末、粒径106 μm 以下のFe粉末および前記希土類酸化物を化学反応式上100%還元するのに要する量(化学量論的必要量)の1.0倍の金属Caをそれぞれ秤量後、それらを混合する。還元剤はCaに限定されず、Mg、CaH₂またはMgH₂が有用である。化学量論的必要量は0.5~2倍が好ましい。化学量論的必要量が0.5倍未満では工業生産上有益な還元/拡散反応が行えず、2倍超では残留する還元剤の量が増大して磁気特性が大きく低下する。次に、混合物をR/D反応炉に入炉後、アルゴン雰囲気中で1000~1300℃×1~10時間加熱するR/D反応を行い、その後室温まで冷却する。R/D反応の加熱条件が1000℃×1時間未満では工業生産上有益なR/D反応が実現されず、1300℃×10時間超ではR/D反応が飽和する。次に、得られたR/D反応生成物を数mmの粒径に粗粉碎後、洗浄する。洗浄により、R/D反応副生成物である未反応の金属Ca、CaO、Ca(OH)₂またはCaCO₃等を洗浄液中に抽出して洗浄液とともに洗い流す。例えば洗浄液として、水をベースにして特開昭63-310905号公報に記載のインヒビター等(水溶性防錆剤等)を5×10⁻³g/l添加した洗浄液を用いると洗浄中または洗浄後の酸化抑制効果を得ることができる。洗浄後に脱水し、続いて工業用アルコール(イソプロピルアルコール等)中に浸漬する。その後脱溶媒し、続いて13.3~1.2×10⁻³Pa(10⁻¹~9×10⁻⁶Torr)の真空中で乾燥してR-T-B系統結磁石用のR/D粉末が得られる。次にR/D粉末を不活性ガ

ス雰囲気中で平均粒径2~10 μm に微粉碎する。次いで、酸化の進行を抑えた条件で磁場で成形後、前記の真空加熱処理を施す。続いて、焼結、熱処理および表面処理を行うことにより本発明の焼結磁石が得られる。表面処理は電解Niめっきまたは無電解Niめっき等の公知の表面処理を適用する。

【0011】以下、実施例により本発明を説明するが、それら実施例により本発明が限定されるものではない。

- (実施例1)母原料としていずれも純度99.9%以上のNd₂O₃、Pr₆O₁₁、Dy₂O₃、FeB、Ga₄Fe₃、Fe粉末(篩分粒径106 μm 以下)を、表1の主要成分組成になるようにそれぞれ適量ずつ秤量後、さらに金属Ca粒(平均粒径数mm)を前記酸化物の還元に必要な化学量論的必要量の1.0倍に秤量した。次に、秤量した各々を混合機に投入して混合した。次に、混合物をR/D反応炉に入炉後、アルゴン雰囲気中で1100℃×4時間加熱してR/D反応を行った後、室温まで冷却した。次に、洗浄液中にR/D反応生成物塊を投入して所定時間洗浄後、脱水した。続いて室温、約6.7Pa(0.05Torr)の真空中で乾燥して、R-Fe-B系R/D粗粉を得た。次にR/D粗粉を32 μm 以下の篩分後、窒素ガスを粉碎媒体とするジェットミルにより平均粒径4 μm に微粉碎した。この微粉の組成は、重量%で、主要成分がNd:23.77%、Pr:6.03%、Dy:2.28%、B:1.10%、Ga:0.12%、残部Feであり、不可避不純物としてCa:0.122%、O:0.550%、C:0.083%を含んでいた。次に、前記微粉を印加磁場強度636.6kA/m(8kOe)、成形圧1.6×10⁸Pa(1.6トン/cm²)で横磁場成形した。次いで、成形体に約6.7×10⁻⁴Pa(5×10⁻⁶Torr)の真空中で1000℃×1時間の真空加熱処理を施した。続いて約1.3×10⁻²Pa(1×10⁻⁴Torr)の真空中で1080℃×2時間の焼結を行った。続いて、アルゴン雰囲気中で900℃×1時間と550℃×1時間の熱処理を行った。熱処理後の焼結体の分析値を表1に、また室温(20℃)で測定した磁気特性を表2に示す。次に、焼結体を所定寸法に加工後、バレル研磨を行った。続いてめっき前処理を施した後、平均膜厚10 μm の電解Niめっきを被覆した。この焼結磁石は実用に耐える良好な耐食性を有していた。
- (比較例1)実施例1の成形体をそのまま、実施例1の真空加熱処理を行わずに焼結した以外は実施例1と同様に焼結磁石を作製した。この焼結体の分析値および磁気特性の測定結果をそれぞれ表1、2に示す。表1、2における実施例1と比較例1との比較から、前記の約6.7×10⁻⁴Pa(5×10⁻⁶Torr)の真空中で1000℃×1時間の真空加熱処理を行った後焼結した実施例1の焼結磁石ではCa含有量が顕著に減少しており、酸素量も低いことがわかる。また、比較例1に比べて、実施例1の焼結磁石は密度、Br、iHc、(BH)_{max}および角型比(Hk/iHc)がいずれも高いことがわかる。

【0012】

【表1】

	Nd (wt%)	Pr (wt%)	Dy (wt%)	B (wt%)	Ga (wt%)	Ca (wt%)	C (wt%)	O (wt%)	Fe (wt%)
実施例 1	23.70	6.00	2.28	1.10	0.12	0.012	0.073	0.540	bal.
実施例 2	23.70	6.00	2.28	1.10	0.12	0.011	0.072	0.500	
実施例 3	19.57	5.33	4.96	0.88	0.08	0.011	0.075	0.198	
比較例 1	23.72	6.01	2.28	1.10	0.12	0.040	0.080	0.570	
比較例 2	23.71	6.00	2.28	1.10	0.12	0.042	0.081	0.580	
比較例 3	19.57	5.34	4.96	0.88	0.08	0.042	0.077	0.350	

【0013】

10 【表2】

	密度 (g/cc)	Br (kG)	iHc (kOe)	(BH) max (MGoe)	Hk/iHc (%)
実施例 1	7.54	12.7	17.8	38.3	95.6
実施例 2	7.55	—	—	—	—
実施例 3	7.63	12.6	23.7	38.6	96.7
比較例 1	7.52	12.6	17.0	37.6	85.0
比較例 2	7.52	—	—	—	—
比較例 3	7.58	12.4	20.0	35.6	87.0

【0014】（実施例2）実施例1で作製した微粉碎粉を用いて、所定の成形装置においてラジアル異方性磁粉を印加しながら圧縮成形して、ラジアル異方性を付与した成形体を作製した。続いて、成形体を約 6.7×10^{-4} Pa (5×10^{-6} Torr) の真空中で $600^{\circ}\text{C} \times 1$ 時間加熱後、さらに約 1.3×10^{-4} Pa (1×10^{-6} Torr) の真空中で $1000^{\circ}\text{C} \times 1$ 時間加熱する2段の真空加熱処理を行った。その後、約 1.3×10^{-2} Pa (1×10^{-4} Torr) の真空中で $1080^{\circ}\text{C} \times 2$ 時間の焼結を行った後、室温まで冷却した。次に、前記焼結体にアルゴン雰囲気中で $900^{\circ}\text{C} \times 1$ 時間と $550^{\circ}\text{C} \times 1$ 時間の熱処理を各1回行った。表1に熱処理後の焼結体の分析値を、表2に密度を示す。次に、焼結肌がなくなるまで加工後、エポキシ樹脂コーティング（平均膜厚 $10 \mu\text{m}$ ）を施して、外径 30mm 、内径 24mm 、軸方向長さ 10mm のラジアル異方性を有する焼結リング磁石を作製した。次に、室温の大気中でこのリング磁石の磁気特性が飽和する条件で外周面の周方向に対称4極着磁を施した後、総磁束量を測定した。その結果、回転機として有用な高い総磁束量が得られた。

（比較例2）実施例1で作製したラジアル異方性を有する成形体をそのまま $1080^{\circ}\text{C} \times 2$ 時間焼結後、室温まで冷却した。以降は実施例2と同様にしてラジアル異方性を有する焼結リング磁石を作製し、評価した。表1に焼結体の分析値を、表2に密度を示す。また、実施例2と同様にして総磁束量を測定した結果、総磁束量は実施例2の焼結リング磁石に比べて8%低かった。不純物として含まれる $\text{Ca}(\text{OH})_2$ が脱水されて CaO になる温度が約 600°C であるので、実施例2のリング磁石が実施例1の焼結磁石に比べて酸素量が低減されているのは、前記 $600^{\circ}\text{C} \times 1$ 時間の1段目の真空加熱処理によって脱離した H_2O に

よる磁粉の酸化が抑制された効果と判断される。

【0015】（実施例3）重量%で主要成分がNd : 19.57%、Pr : 5.33%、Dy : 4.96%、B : 0.88%、Ga : 0.08%、残部FeのR/D粉末になるように母原料および金属Ca（化学量論的必要量の1.1倍）を適量比率で秤量し、混合した以外は実施例1と同様にしてR/D反応、洗浄および乾燥を行った。得られたR/D粗粉を用いて窒素ガスを粉碎媒体としてジェットミル微粉碎し、平均粒径 $3.3 \mu\text{m}$ の微粉を得た。次に、前記微粉を大気に触れさせずにジェットミルの微粉排出口に設置した鉱油油（商品名：出光興産製出光スーパーMPA-30）中に直接回収してスラリー化した。次に、このスラリーを用いて、印加磁場強度 795.8kA/m (10kOe)、成形圧力 7.8×10^7 Pa (0.8ton/cm^2) の条件で横磁場湿式成形を行い、略扁平扇形状の成形体を得た。次に、成形体を真空加熱炉に投入後、約 6.7Pa (5×10^{-2} Torr) の真空中で $200^{\circ}\text{C} \times 2$ 時間加熱する脱油処理を施した。引き続き約 6.7×10^{-4} Pa (5×10^{-6} Torr) の真空中で $600^{\circ}\text{C} \times 1$ 時間の1段目の真空加熱処理を行った。続いて約 6.7×10^{-4} Pa (5×10^{-6} Torr) の真空中で $1000^{\circ}\text{C} \times 1$ 時間の2段目の真空加熱処理を施した。続いて、約 1.3×10^{-2} Pa (1×10^{-4} Torr) の真空中で $1070^{\circ}\text{C} \times 3$ 時間加熱後室温まで冷却して焼結体を得た。次に、所定形状に加工後、アルゴン雰囲気中で $900^{\circ}\text{C} \times 1$ 時間と $550^{\circ}\text{C} \times 1$ 時間の熱処理を各1回施し、室温まで冷却した。次に、パレル研磨を行い、その後めっき前処理を行った。続いて電解Niめっき（平均膜厚 $10 \mu\text{m}$ ）を被覆して本発明の焼結磁石を得た。表1に前記焼結体の分析値を、表2に磁気特性および密度を示す。次に、作製した一対の扁平な扇形状焼結磁石 22.22 （厚み $t_m = 4\text{mm}$ ）を、磁気特性が

飽和する条件で着磁後、磁気空隙40を介して強磁性体製のヨーク23、25の表面に各々接着して図4のボイスコイルモータ20を作製した。次に、ボイスコイルモータ20の磁気空隙40において、 $(tg/2)$ でかつ空隙磁束密度がピーク値を示す点Pの空隙磁束密度を測定したところ、ボイスコイルモータとして有用な空隙磁束密度が得られた。

(比較例3) 実施例3で作製した略扁平扇形状の成形体をそのまま約 1.3×10^{-2} Pa (1×10^{-4} Torr)の真空中で $1070^{\circ}\text{C} \times 3$ 時間加熱後室温まで冷却して焼結体を得た。以降は実施例3と同様にして扁平扇形状焼結磁石(厚み $t_m = 4$ mm)を作製した。この比較例の焼結磁石を、実施例3の扁平扇形状焼結磁石22、22に替えて図4のボイスコイルモータ50に組み込んだ。次に、このボイスコイルモータにおいて、 $(tg/2)$ でかつ空隙磁束密度がピーク値を示す位置の空隙磁束密度を測定した。その結果、実施例3のボイスコイルモータに比べて空隙磁束密度のピーク値が5%低かった。実施例3と比較例3の比較から、酸化の進行を阻止できる低酸素プロセス(湿式成形プロセス)および前記真空加熱処理を施すことにより、酸素量がさらに低減されて $R_2Fe_{14}B$ 型金属間化合物(主相)に相当する高密度になり、さらにボイスコイルモータの空隙磁束密度を向上できることがわかる。実施例3では鉱物油を用いた場合を記載したが、合成油または植物油を用いてもよい。あるいは鉱物油と合成油、合成油と植物油、鉱物油と合成油と植物油との混合物を用いてもよい。実施例3に記載の湿式成形プロセスと前記真空加熱処理とを併用することにより、重量%で、R/D合金粉末を100%配合してなるR-T-B系焼結磁石の酸素量を0.2%未満、Ca含有量を0.02%未満(0を含まず)、炭素含有量を0.1%以下および密度を $7.57 \sim 7.70 \text{ Mg/m}^3$ (g/cm^3)にすることができる。同時に、 20°C において、 $302.5 \sim 398 \text{ kJ/m}^3$ ($38 \sim 50 \text{ MG0e}$)の(BH)_{max}、 $1432.4 \sim 1989.5 \text{ kA/m}$ ($18 \sim 25 \text{ kOe}$)のiHcおよび $95 \sim 98\%$ の(Hk/iHc)を実現することができる。

【0016】(実施例4) 実施例1～3および比較例1～3の各焼結磁石からそれぞれ厚み1 mmの板状試料を切り出した後、ラッピング加工により厚み $100 \mu\text{m}$ に研磨した。続いて、Arイオンミリング等により厚み約 $0.1 \mu\text{m}$ に薄膜化した。次に、(株)日立製作所製の透過型電子顕微鏡FETEM(商品名:HF-2100)に前記各薄膜試料を順次セットした後、加速電圧200kV、フィラメント電流 $50 \mu\text{A}$ 、分解能19nmの条件で各薄膜試料の断面組織を観察し、断面写真を撮影した。また、観察した対象視野の組成の同定はEDX分析装置(NORAN社製、商品名:VANTAGE)を用い、ビーム径 0.7 nm の条件で行った。図1は実施例1の焼結磁石の代表的な主相結晶粒部分を示す断面組織を前記透過型電子顕微鏡により撮影した写真である。図2は図1に対応する模式図である。電子回折結果等から1、21は芯部の主相結晶粒であり、5、25

はそれぞれ平均結晶粒径が $0.5 \sim 50 \text{ nm}$ の範囲にある複数の主相結晶粒からなる表層部であり、13は希土類リッチ相であることがわかった。希土類リッチ相13(点B等)を介して主相結晶粒部分10、30が存在する。表層部5の点C、点Dおよび点Eと、芯部1の点Aおよび芯部21の点Fで電子回折を行い、各測定位置間のc軸方位差を測定した。結果を表3に示す。表3において、A-Cとは点Aと点Cとのc軸方位差を意味する。表3より、芯部1(点A)のc軸方位と表層部5の点C、点Dおよび点Eの各主相結晶粒のc軸方位とのなす角度は $5 \sim 76^{\circ}$ であった。この結果から、表層部2を構成する主相結晶粒はランダムなc軸方位を有することがわかる。また希土類リッチ相13を介して存在する芯部1と芯部21とのc軸方位差は 1° 以内であった。このことは、磁場中圧縮成形時の個々の微粉粒子毎で最も大粒径の芯部の主相結晶粒が印加磁場方向に配向した効果を示すものと判断される。

【0017】

【表3】

	c軸方位差(度)
A-C	5
A-D	32
A-E	76
A-F	1

【0018】実施例1の焼結磁石の断面組織には図1、2以外のマイクロ組織が観察された。図3に、実施例1の焼結磁石のマイクロ組織の特徴を模式的に示す。図3では、希土類リッチ相77を介して主相結晶粒部分60、70および80が存在する。主相結晶粒部分60は図1、2と同様のマイクロ組織である。61は芯部の主相結晶粒、65aは芯部61を覆う複数の微細な主相結晶粒からなる表層部である。主相結晶粒部分70は芯部の主相結晶粒71と、芯部71の表層部に点在する微細な主相結晶粒72とからなる。主相結晶粒部分80は1個の主相結晶粒85のみからなる。また、実施例1の焼結磁石では、主相結晶粒部分を1個とカウントしたとき、観察した100個の主相結晶粒部分あたり、図3の65型の主相結晶粒部分が6個、図3の70型の主相結晶粒部分が11個、主相結晶粒部分80型が83個観察された。次に、前記と同様にして実施例2、3および比較例1～3の焼結磁石の断面組織をそれぞれ撮影し、評価した。それぞれの断面写真において、各主相結晶粒部分を1個とカウントしたとき、観察した100個の主相結晶粒部分あたり、芯部および表層部の複数の主相結晶粒からなる主相結晶粒部分の個数比率を測定した。結果を表4に示す。表4より、実施例1～3の焼結磁石では、芯部および表層部の複数の主相結晶粒からなる主相結晶粒部分の個数比率が50%未満であることがわかる。

【0019】

【表4】

	(芯部+表層部)/(主相結晶粒部分) の個数比率(%)
実施例 1	24
実施例 2	17
実施例 3	7
比較例 1	55
比較例 2	58
比較例 3	51

【0020】上記実施例ではボイスコイルモータを記載したが、本発明はこれに限定されず、有用なスピンドルモータあるいはリニアモータを構成することができる。

【0021】本発明はR/D粉末のみを用いて製造したR-T-B系統結磁石に限定されず、R/D粉末と溶製合金粉末とを所定比率で配合してなるR-T-B系統結磁石を包含する。この場合、原料原価を低減するために、(R/D粉末):(溶製合金粉末)は、10~100重量部:90~0重量部が好ましく、30~100重量部:70~0重量部がより好ましく、50~100重量部:50~0重量部が特に好ましい。

【0022】上記実施例では還元剤に金属Caを用いたが、Caの水素化物、金属Mg、Mgの水素化物またはそれらの混合物を用いた場合でも、Mg含有量または(Ca+Mg)含有量を0.02重量%未満(0を含まず)にできるとともに、上記特定量範囲の酸素量、炭素量およびマイクロ組織を有する焼結磁石を提供することができる。

【0023】上記実施例ではラジアル異方性を有するリング磁石を記載したが、本発明はこれに限定されない。磁極数が4~32極の表面多極異方性を有するR-T-B

系統結リング磁石または径2極異方性を有するR-T-B系統結リング磁石を作製した場合、従来の還元/拡散法によるR-T-B系統結磁石用合金粉末を用いた場合に比べて顕著に高い磁気特性を実現可能である。

【0024】

【発明の効果】以上記述の通り、本発明によれば、還元拡散法によるR-T-B系統結磁石用合金粉末を用いて製造された安価でかつ高性能のR-T-B系統結磁石、リング磁石およびボイスコイルモータを提供することができる。

【図面の簡単な説明】

【図1】本発明の焼結磁石の代表的な断面組織を透過型電子顕微鏡により撮影した写真である。

【図2】図1を説明する模式図である。

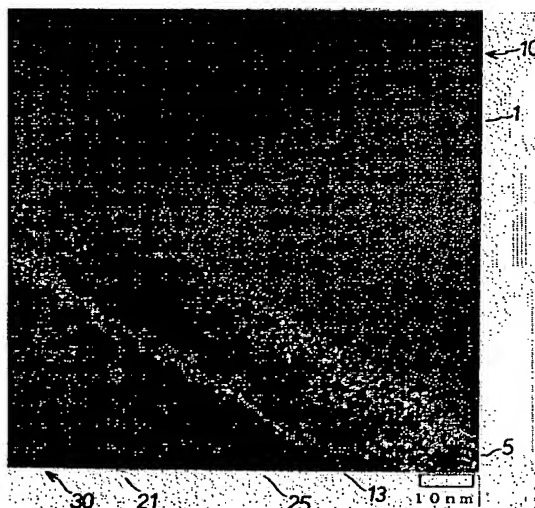
【図3】本発明の焼結磁石のマイクロ組織を説明する模式図である。

【図4】本発明のボイスコイルモータの要部断面図(a)、(a)のA-A線矢視断面図(b)である。

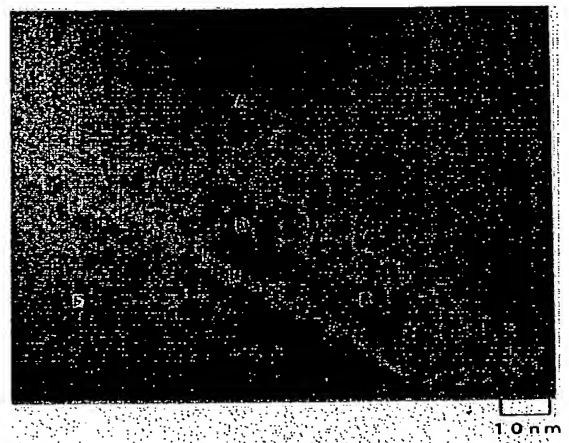
【符号の説明】

- 1, 21, 61, 71, 85 芯部、5, 25, 65, 72 表層部、10, 30, 60, 70, 80 主相結晶粒部分、13, 77 希土類リッチ相、20 磁極境界、22 扁平状磁石、23 強磁性体製の上ヨーク、24 強磁性体製の支柱、25 強磁性体製の下ヨーク、40 磁気空隙、26 可動コイル、27 アーム、30 軸、31 可動コイルの中心と軸の中心とを結ぶ直線、32 軸の中心と磁極境界に沿う直線とを結んだ直線、50 ボイスコイルモータ、65a 主相微結晶粒。

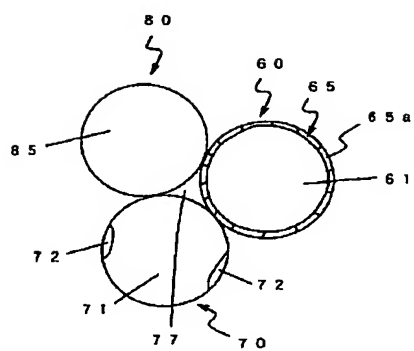
【図1】



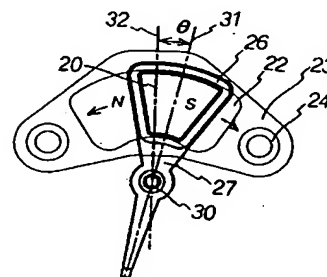
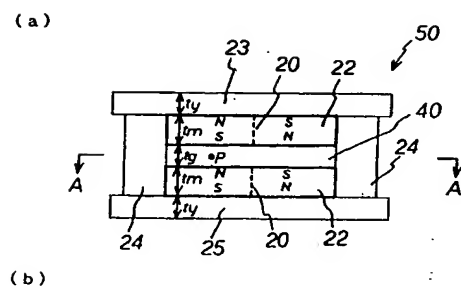
【図2】



【図 3】



【図 4】



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113685

(22)Date of filing : 14.04.2000 (72)Inventor : FUJIMORI NOBUHIKO
KAWADA TSUNEHIO

(54) R-T-B SINTERED MAGNET, RING MAGNET AND VOICE COIL MOTOR



(57)Abstract:

PROBLEM TO BE SOLVED: To provide an R-T-B sintered magnet which is manufactured by the use of R-T-B sintered magnet alloy powder through a reduction diffusion method, inexpensive, and high in performance, a ring magnet and a voice coil motor.

SOLUTION: An R-T-B sintered magnet contains an R₂T₁₄B intermetallic compound (R denotes one or more rare earth elements, including Y; and T is Fe or Fe and Co) as a main phase, is below 0.02 wt.% (not zero) in avoidable Ca content, and provided with a main phase crystal grain part which resides through a rare earth rich phase, containing at least two or more parts that are a core part and a surface both formed of main phase crystal grains, where the main phase crystal grains of the surface are 0.5 to 50 nm in average crystal grain diameter.

LEGAL STATUS

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registration]

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[Patent number]

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CLAIMS

[Claim(s)]

[Claim 1] R₂T₁₄B mold intermetallic compound (R is one sort of the rare earth elements containing Y, or two sorts or more) It is the R-T-B system sintered magnet made into the main phase. T -- Fe, or Fe and Co -- it is -- The amount of calcium contained is less than (0 is not included) 0.02 % of the weight. The R-T-B system sintered magnet characterized by the diameter of average crystal grain of the main phase crystal grain of said surface section being 0.5-50nm while what the main phase crystal grain part which exists through a rare earth rich phase becomes from at least two or more main phase crystal grain, a core part and the surface section, is included.

[Claim 2] The rare earth sintered magnet according to claim 1 which is less than

50% per main phase crystal grain part whose number ratios which the main phase crystal grain part which consists of an aforementioned core part and the aforementioned surface section occupies when the main phase crystal grain part which exists through a rare earth rich phase is counted among one piece are 100 pieces.

[Claim 3] weight % -- a major component -- R:27 - 33%, B:0.8 - 1.5%, and M:0.01 - 1% (M -- aluminum --) The amount of oxygen which consists of one sort of Ga, Nb, and Cu or two sorts or more, and the remainder T, and is contained impossibly Less than 0.6%, The R-T-B system sintered magnet according to claim 1 or 2 0.2% or less and whose consistency a carbon content is three or more 7.53 g/cm, whose square shape ratio (Hk/iHc) in 20 degrees C is 95% or more and whose maximum energy product (BH) max is more than 302.5 kJ/m³ (38MGOe).

[Claim 4] R2T14B mold intermetallic compound (R is one sort of the rare earth elements containing Y, or two sorts or more) It is the ring magnet which consists of a R-T-B system sintered magnet made into the main phase. T -- Fe, or Fe and Co -- it is -- The amount of calcium contained impossibly is less than (0 is not included) 0.02 % of the weight. The ring magnet characterized by the diameter of average crystal grain of the main phase crystal grain of said surface section being 0.5-50nm while what the main phase crystal grain part which exists through a rare earth rich phase becomes from at least two or more main phase crystal grain, a core part and the surface section, is included.

[Claim 5] R2T14B mold intermetallic compound (R is one sort of the rare earth elements containing Y, or two sorts or more) It is a voice coil motor using the R-T-B system sintered magnet made into the main phase. T -- Fe, or Fe and Co -- it is -- Said sintered magnet contains what the main phase crystal grain part which exists through a rare earth rich phase while the amount of calcium contained impossibly is less than (0 is not included) 0.02 % of the weight becomes from at least two or more main phase crystal grain, a core part and the surface section.

And the voice coil motor characterized by the diameter of average crystal grain of the main phase crystal grain of said surface section being 0.5-50nm.

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] this invention was manufactured using the alloy powder for R-T-B system sintered magnets by the reduction diffusion method (R is one sort in the rare earth elements containing Y, or two sorts or more, and T is Fe, or Fe and Co) -- it is cheap and is related with the R-T-B system rare earth sintered magnet, ring magnet, and voice coil motor of high performance.

[0002]

[Description of the Prior Art] Need of the R-T-B system sintered magnet

[comparatively / in resource / abundant rare earth elements, such as Nd, Dy, or Pr, and] which uses Fe or Fe, and Co and B as a major component increases every year from having the high magnetic properties which are not in the former, and it came to occupy most rare earth magnet commercial scenes in the present condition. In connection with the increase of this need, the cost reduction of a R-

T-B system sintered magnet progresses, it is more cheap and the thing of high performance has been required.

[0003] In the alloy powder adjusted to the R-T-B system sintered magnet presentation, surface treatment is pressed, sintered, heat-treated and carried out, and a R-T-B system sintered magnet is manufactured. The grinding powder of R-T-B system ingot alloys (a strip cast alloy, or the RF dissolution / casting alloy) is mainly used as R-T-B system alloy powder. On the other hand, by blending the metal calcium which are a rare earth oxide, Fe-B (Fe-Co-B) alloy powder, Fe powder, and a reducing agent by the optimum dose ratio, and heating it in an inert gas ambient atmosphere after mixing as cheap R-T-B system alloy powder, calcium reduction / diffusion reaction is performed and it cools to a room temperature after that. Then, the process which washes and dries [vacuum (heating)]: There is alloy powder (this is hereafter called R/D powder) produced by reduction/diffusion method (Reduction/Diffusion law).

[0004] However, compared with the R-T-B system sintered magnet produced using ingot alloy powder, the R-T-B system sintered magnet produced using R/D powder has the problem that magnetic properties, such as a square shape ratio and coercive force iH_c , are low. This is judged because CaO, oxygen, carbon, etc. are included so much impossibly. In order to reduce said unescapable impurity, the reaction generation lump after calcium reduction / diffusion reaction is thrown in in penetrant removers (water etc.), and the various devices which extract reaction by-products, such as CaO, efficiently and flush them in a penetrant remover are given. Moreover, when it adds mechanical grinding or washing time amount is extended, and oxidation advances during washing or it is exposed to atmospheric air from the vacuum or inert gas ambient atmosphere at the time of desiccation, there is [in order to make easy to extract in a penetrant remover the reaction by-product which intervenes in a R/D reaction main constituent,] a problem that oxidation advances notably.

[0005] As a measure for the above-mentioned problem, rinsing, dehydration, and the approach of carrying out a vacuum drying are indicated by JP,63-310905,A

using the water which carried out 10-3-10-2 g/l addition of the inhibitor (corrosion inhibitor) in rinsing of the resultant after calcium reduction / diffusion reaction as hypoxia and the manufacture approach of obtaining the R-T-B system R/D powder of low calcium. However, even when this manufacture approach was adopted, as for calcium content of industrial production top R-T-B system R/D powder, examination of this invention persons showed that reduction to about at most 0.05 - 0.1 % of the weight was a limitation. For this reason, when a R-T-B system sintered magnet is produced only using said R/D powder, as compared with the R-T-B system sintered magnet produced using R-T-B system ingot alloy powder (calcium content: about 0 ppm), calcium content becomes very high. There is a problem that a square shape ratio (H_k/iH_c) deteriorates in coincidence, and it cannot apply to a heat-resistant application at it. H_k is the ** value of H (magnetic field strength) shaft of the location where $4\pi i$ (intensity of magnetization) becomes $0.9B_r(s)$ in a $4\pi i$ - H demagnetization curve. B_r is a residual magnetic flux density. Said problem is common in the R-T-B system sintered magnet which carried out specified quantity (for example, 10 - 100 % of the weight) combination, and produced the R/D alloy powder for the conventional R-T-B system sintered magnets.

[0006]

[Problem(s) to be Solved by the Invention] Therefore, it is cheap and the technical problem of this invention is a thing which was manufactured using the alloy powder for R-T-B system sintered magnets by the reduction diffusion method (R is one sort in the rare earth elements containing Y, or two sorts or more, and T is Fe, or Fe and Co) and which offer the R-T-B system sintered magnet, ring magnet, and voice coil motor of high performance.

[0007]

[Means for Solving the Problem] This invention which solved the above-mentioned technical problem is an R2T14B mold intermetallic compound (R is one sort of the rare earth elements containing Y, or two sorts or more). It is the R-T-B system sintered magnet made into the main phase. T -- Fe, or Fe and Co -

- it is -- The amount of calcium contained is less than (0 is not included) 0.02 % of the weight. While what the main phase crystal grain part which exists through a rare earth rich phase becomes from at least two or more main phase crystal grain, a core part and the surface section, is included, the diameter of average crystal grain of the main phase crystal grain of said surface section is the R-T-B system sintered magnet which is 0.5-50nm. While having said calcium content, when the main phase crystal grain part consists of at least two or more main phase crystal grain, a core part and the surface section, compared with the case where the alloy powder for R-T-B system sintered magnets by the above-mentioned conventional reduction diffusion method is used, a square shape ratio (H_k/iH_c), coercive force iH_c , etc. can be improved notably. Moreover, when the main phase crystal grain part which exists through a rare earth rich phase is counted among one piece in said sintered magnet, When the number ratio which the main phase crystal grain part which consists of an aforementioned core part and the aforementioned surface section occupies is less than 50% in the main phase crystal grain part whose number is 100 The R-T-B system sintered magnet which improved notably a square shape ratio (H_k/iH_c), coercive force iH_c , etc. compared with the case where the alloy powder for R-T-B system sintered magnets by the above-mentioned conventional reduction diffusion method is used is obtained. Said sintered magnet is weight %.

A major component presentation Moreover, R:27 - 33%, B:0.8 - 1.5%, and M:0.01 - 1% (it Ga(s) and Nb(s) M -- aluminum --) When less than 0.6% and a carbon content are [0.2% or less and a consistency] three or more 7.53 g/cm, the amount of oxygen which consists of one sort of Cu or two sorts or more, and the remainder T, and is contained is The square shape ratio (H_k/iH_c) in 20 degrees C becomes 95% or more, maximum energy product (BH) max becomes more than 302.5 kJ/m³ (38MGOe), and it is desirable.

[0008] On the occasion of manufacture of the sintered magnet of this invention, the vacuum heating conditions carried out to a Plastic solid indispensable are explained below. First, the alloy powder for R-T-B system sintered magnets

manufactured by the reduction diffusion method is fabricated among [after grinding to predetermined particle size] a magnetic field, and a Plastic solid is acquired. Next, heat treatment for decalcium which heats a Plastic solid at 850-1050 degrees C in the vacuum of 133 to 1.2×10^{-3} Pa (1×10^{-2} - 9×10^{-6} Torr) is performed. The 1st step of vacuum heat-treatment especially heated to said Plastic solid at 550-650 degrees C in the vacuum of 133 to 1.2×10^{-3} Pa (1×10^{-2} - 9×10^{-6} Torr), Then, by performing the 2nd step of vacuum heat-treatment heated at 850-1050 degrees C in the vacuum of 133 to 1.2×10^{-3} Pa (1×10^{-2} - 9×10^{-6} Torr), since the amount of oxygen is reduced while decalcium becomes remarkable, it is desirable. vacuum heat-treatment conditions -- the case of one step of vacuum heat-treatment -- the degree of vacuum of 133 to 1.2×10^{-3} Pa (1×10^{-2} - 9×10^{-6} Torr) -- and it is desirable to choose whenever [stoving temperature / of 850-1050 degrees C] because of decalcium. calcium removed by this vacuum heat-treatment is considered to be the part which exists in the state of Metal calcium. That is, whenever [1 step of stoving temperature / of vacuum heat-treatment] has desirable 1050 degrees C or less with it. [more than the good and melting point (851 degrees C) of Metal calcium, and] [lower than sintering temperature] In 1050-degree-C **, the eburation by sintering progresses rapidly and decalcium is because it is difficult. The range of one step of still more desirable vacuum heat-treatment temperature is 900-1000 degrees C. The heating time of one step of vacuum heat-treatment has 0.5 - 5 desirable hours. Decalcium is not fully performed, a decalcium reaction is saturated with ** for 5 hours, and heat treatment cost increases in less than 0.5 hours. If a degree of vacuum is 133 to 1.2×10^{-3} Pa on industrial production (1×10^{-2} - 9×10^{-6} Torr), it is enough to realize decalcium. Decalcium is difficult and adoption of the high vacuum of ** (9×10^{-6} Torr) is lacking in practicality 1.2×10^{-3} Pa at under 133Pa (1×10^{-2} Torr). the case of two steps of vacuum heat-treatment -- as the 1st step of vacuum heat-treatment conditions -- the inside of the vacuum of 133 to 1.2×10^{-3} Pa (1×10^{-2} - 9×10^{-6} Torr) -- and it is desirable especially from the point of reduction of the amount of oxygen to adopt 550-650-degree C heating conditions.

It is judged that this has that the temperature which calcium (OH)₂ which is a reaction by-product divides into CaO and H₂O is near 580 degree C, and correlation. Examination of this invention persons showed that the oxidation at the time of decalcium was suppressed, and the amount of oxygen could be reduced if the conditions of the 1st step of vacuum heat-treatment are set up so that it may dehydration-ize at about 580 degrees C. Less than 550 degrees C of dehydration-izing are insufficient, and the inclination for oxidation resulting from the pyrolysis of a reaction by-product to become remarkable was accepted in 650-degree-C **. Heating maintenance of the 1st step of vacuum heat-treatment has 0.5 - 5 good hours. The reason for limitation of the 2nd step of vacuum heat-treatment conditions is the same as that of the above.

[0009] The reason for presentation limitation of the R-T-B system sintered magnet of this invention is explained below. Hereafter, it is weight % which is only being described as %. 27 - 33% of the amount of R is desirable. The implementation of iHc whose amount of R is equal to practical use at less than 27% is difficult, and Br falls greatly by ** 33%. It is chosen so that at least one sort of Nd, Dy, and Pr may be practically included as R. 0.8 - 1.5% of the amount of B is desirable, and is more desirable. [0.9 - 1.2% of] The implementation of iHc whose amount of B is equal to practical use at less than 0.8% is difficult, and Br falls greatly by ** 1.5%. 0.01 - 1% of the amount of Nb(s) is desirable. At less than 0.01%, depressor effect [as opposed to coarsening at the time of sintering in the amount of Nb(s)] is not acquired, but Br falls greatly by ** 1%. 0.01 - 1% of the amount of aluminum is desirable. At less than 0.01%, the improvement effectiveness of iHc is not acquired for the amount of aluminum, but Br falls greatly by ** 1%. 0.01 - 1% of the amount of Ga(s) is desirable. At less than 0.01%, the improvement effectiveness of iHc is not acquired for the amount of Ga(s), but Br falls greatly by ** 1%. 0.01 - 1% of the amount of Cu(s) is desirable. Although minute amount addition of Cu brings about improvement in iHc, effectiveness is not accepted at less than 0.01%, but the improvement effectiveness of iHc is saturated with ** 1%. 0.3 - 5% of the amount of Co(es) is

desirable. At less than 0.3%, the Curie point and the corrosion-resistant improvement effectiveness are not acquired for the amount of Co(es), but Br and iHc fall greatly by ** 5%. In order to realize the R-T-B system sintered magnet and ring magnet which bear heat-resistant applications, such as a rotating machine or a voice coil motor, less than 0.6% of the amount of oxygen contained is desirable, is more desirable, and is desirable. [especially less than 0.2% of] [0.4% or less of] Furthermore, when the amount of oxygen is reduced to 0.3% or less, R content is preferably good to consider as 29 - 31% more preferably 28 to 32%. Since rare earth carbide increases by the increment in a carbon content and magnetic properties fall, a carbon content is more preferably [0.1% or less of] good 0.2% or less preferably.

[0010]

[Embodiment of the Invention] Desirable manufacture conditions other than the vacuum-heat-treatment condition concerning this invention are explained. They are mixed for the 1.0 times as many metal calcium as the amount (stoichiometric initial complement) taken to return the oxide (Nd, Dy) powder of 99.9% or more of purity, Fe-B powder, Fe powder of a particle-size the undershirt of 106 micrometers, and said rare earth oxide 100% on a reaction formula after weighing capacity, respectively so that a major component may serve as Remainder Fe R (Nd, Dy):30%, B:1.0%, and aluminum:0.03%. A reducing agent is not limited to calcium but Mg, CaH₂, or MgH₂ are useful. A stoichiometric initial complement has 0.5 to 2 desirable times. A stoichiometric initial complement cannot perform useful reduction/diffusion reaction on industrial production in less than 0.5 times, but in 2 double **, the amount of the reducing agent which remains increases and magnetic properties fall greatly. Next, the R/D reaction which heats mixture in an argon ambient atmosphere behind an ON furnace to a R/D fission reactor for 1000-1300 degree-Cx 1 to 10 hours is performed, and it cools to a room temperature after that. Less than [1000 degree-Cx1 hour], a useful R/D reaction is not realized for the heating conditions of a R/D reaction on industrial production, but a R/D reaction is saturated with **

for 1300 degree-Cx 10 hours. Next, the acquired R/D resultant is washed after coarse grinding in particle size of several mm. By washing, the unreacted metals (OH) calcium [calcium, CaO and] 2 or CaCO₃ grade which is a R/D reaction by-product is extracted in a penetrant remover, and it washes away with a penetrant remover. For example, as a penetrant remover, water is used as the base, and if the penetrant remover which carried out 5x10⁻³ g/l addition of the inhibitor of a publication etc. (water-soluble rust-proofer etc.) is used for JP,63-310905,A, the oxidation depressor effect under washing or after washing can be acquired. It is continuously dehydrated and immersed into industrial alcohol (isopropyl alcohol etc.) after washing. It deliquors after that, and it dries continuously in the vacuum of 13.3 to 1.2x10 to 3 Pa (10⁻¹ - 9x10⁻⁶Torr), and the R/D powder for R-T-B system sintered magnets is obtained. Next, R/D powder is pulverized in mean particle diameter of 2-10 micrometers in an inert gas ambient atmosphere. Subsequently, the aforementioned vacuum heat-treatment is performed after shaping among a magnetic field on the conditions which suppressed advance of oxidation. Then, the sintered magnet of this invention is obtained by performing sintering, heat treatment, and surface treatment. Surface treatment applies well-known surface treatment, such as electrolysis nickel plating or non-electrolyzed nickel plating.

[0011] Hereafter, this invention is not limited by these examples although an example explains this invention. (Example 1) as a mother raw material -- each -- Nd₂ of 99.9% or more of purity -- O₃ and Pr₆ -- O₁₁ and Dy₂ -- the weighing capacity of the metal calcium grain (mean particle diameter of several mm) was further increased 1.0 times of a stoichiometric initial complement required for reduction of said oxide after optimum dose [every] weighing capacity, respectively so that it might become the major component presentation of Table 1 about O₃, FeB, Ga₄Fe₃, and Fe powder (screen analysis particle-size undershirt of 106 micrometers). Next, each which carried out weighing capacity was supplied to the mixer, and it mixed. Next, after heating mixture in argon **** behind the ON furnace to the R/D fission reactor for 1100 degree-Cx 4 hours and

performing a R/D reaction, it cooled to the room temperature. Next, the R/D resultant lump was thrown in in the penetrant remover, and it dehydrated after predetermined time washing. Then, it dried in the room temperature and about 6.7Pa (0.05Torr) vacuum, and R-Fe-B system R/D coarse powder was obtained. Next, it pulverized in mean particle diameter of 4 micrometers with the jet mill which uses R/D coarse powder the screen analysis back, and uses nitrogen gas as tumbling media at a 32-mesh undershirt. It is weight %, major components are Nd:23.77%, Pr:6.03%, Dy:2.28%, B:1.10%, Ga:0.12%, and Remainder Fe, and the presentation of these fines contained calcium:0.122%, O:0.550%, and C:0.083% as an unescapable impurity. Next, horizontal magnetic field shaping of said fines was carried out with impression magnetic-field-strength 636.6 kA/m (8kOe) and the moulding pressure of 1.6×10^8 Pa (1.6 t/cm²). Subsequently, vacuum heat-treatment of 1000 degree-Cx 1 hour was performed to the Plastic solid in the vacuum of about 6.7×10^{-6} to 4 Pa (5×10^{-6} Torr). Then, sintering of 1080 degree-Cx 2 hours was performed in the vacuum of about 1.3×10^{-4} to 2 Pa (1×10^{-4} Torr). Then, heat treatment of 900 degree-Cx 1 hour, and 550 degree-Cx 1 hour was performed in the argon ambient atmosphere. The magnetic properties which measured the analysis value of the sintered compact after heat treatment at the room temperature (20 degrees C) to Table 1 again are shown in Table 2. Next, barrel finishing was performed after processing a sintered compact into a predetermined dimension. Then, after performing plating pretreatment, the electrolysis nickel plating of 10 micrometers of average thickness was covered. This sintered magnet had the good corrosion resistance which is equal to practical use.

(Example 1 of a comparison) The sintered magnet was produced like the example 1 except having sintered the Plastic solid of an example 1 as it was, without performing vacuum heat-treatment of an example 1. The analysis value of this sintered compact and the measurement result of magnetic properties are shown in Tables 1 and 2, respectively. As for the comparison with the example 1 and the example 1 of a comparison in Tables 1 and 2, in the sintered magnet of

the example 1 which performed vacuum heat-treatment of 1000 degree-Cx 1 hour in the vacuum of the about 6.7×10^{-4} Pa (5×10^{-6} Torr) and which carried out the afterbaking join, calcium content is decreasing notably and that it is low also shows the amount of oxygen. Moreover, compared with the example 1 of a comparison, the sintered magnet of an example 1 is understood that each a consistency, Br and iHc, (BH) max, and a square shape ratio (Hk/iHc) are high.

[0012]

[Table 1]

	Nd (wt%)	Pr (wt%)	Dy (wt%)	B (wt%)	Ga (wt%)	Ca (wt%)	C (wt%)	O (wt%)	Fe (wt%)
実施例 1	23.70	6.00	2.28	1.10	0.12	0.012	0.073	0.540	bal.
実施例 2	23.70	6.00	2.28	1.10	0.12	0.011	0.072	0.500	
実施例 3	19.57	5.33	4.96	0.88	0.08	0.011	0.075	0.198	
比較例 1	23.72	6.01	2.28	1.10	0.12	0.040	0.080	0.570	
比較例 2	23.71	6.00	2.28	1.10	0.12	0.042	0.081	0.580	
比較例 3	19.57	5.34	4.96	0.88	0.08	0.042	0.077	0.350	

[0013]

[Table 2]

	密度 (g/cc)	Br (kG)	iHc (kOe)	(BH) max (MGOe)	Hk/iHc (%)
実施例 1	7.54	12.7	17.8	38.3	95.6
実施例 2	7.55	—	—	—	—
実施例 3	7.63	12.6	23.7	38.6	96.7
比較例 1	7.52	12.6	17.0	37.6	85.0
比較例 2	7.52	—	—	—	—
比較例 3	7.58	12.4	20.0	35.6	87.0

[0014] (Example 2) It pressed using the pulverizing powder produced in the example 1, impressing a radial anisotropy magnetic field in predetermined shaping equipment, and the Plastic solid which gave the radial anisotropy was produced. Then, two steps of vacuum heat-treatment which heats a Plastic solid further after 600 degree-Cx 1-hour heating for 1000 degree-Cx 1 hour in the vacuum of about 1.3×10^{-4} Pa (1×10^{-6} Torr) in the vacuum of about 6.7×10^{-4} Pa.

Pa (5×10^{-6} Torr) was performed. Then, after performing sintering of 1080 degree-Cx 2 hours in the vacuum of about 1.3×10 to 2 Pa (1×10^{-4} Torr), it cooled to the room temperature. Next, heat treatment of 900 degree-Cx 1 hour, and 550 degree-Cx 1 hour was performed to said sintered compact once each in the argon ambient atmosphere. A consistency is shown for the analysis value of the sintered compact after heat-treating to Table 1 in Table 2. Next, epoxy resin coating (10 micrometers of average thickness) was performed after processing until the sintering skin was lost, and the sintered ring magnet which has a radial anisotropy with the outer diameter of 30mm, a bore [of 24mm], and a shaft-orientations die length of 10mm was produced. Next, after giving symmetry 4 pole magnetization to the hoop direction of a peripheral face on the conditions on which the magnetic properties of this ring magnet are saturated in the atmospheric air of a room temperature, the amount of total magnetic flux was measured. Consequently, the high amount of total magnetic flux useful as a rotating machine was obtained.

(Example 2 of a comparison) The Plastic solid which has the radial anisotropy produced in the example 1 was cooled to the room temperature after 1080 degree-Cx 2-hour sintering as it was. The sintered ring magnet which has a radial anisotropy like an example 2 was produced and evaluated henceforth. The analysis value of a sintered compact is shown in Table 1, and a consistency is shown in Table 2. Moreover, as a result of measuring the amount of total magnetic flux like an example 2, the amount of total magnetic flux was low 8% compared with the sintered ring magnet of an example 2. Since the temperature which calcium (OH)₂ contained as an impurity is dehydrated, and is set to CaO is about 600 degrees C, the ring magnet of an example 2 is judged to be the effectiveness that oxidation of the magnetic powder by H₂O desorbed from the amount of oxygen being reduced compared with the sintered magnet of an example 1 by the 1st step of vacuum heat-treatment of said 600 degree-Cx 1 hour was controlled.

[0015] (Example 3) Weighing capacity of a mother raw material and the metal

calcium (1.1 times of a stoichiometric initial complement) was carried out by the optimum dose ratio, and R/D reaction, washing, and desiccation were performed like the example 1 except having mixed so that a major component might become the R/D powder of Remainder Fe by weight % Nd:19.57%, Pr:5.33%, Dy:4.96%, B:0.88%, and Ga:0.08%. Using the obtained R/D coarse powder, by using nitrogen gas as tumbling media, jet mill pulverizing was carried out and fines with a mean particle diameter of 3.3 micrometers were obtained. Next, in the straight mineral oil (trade name: Idemitsu Kosan production light super sol PA-30) which installed said fines in the fines exhaust port of a jet mill, without making atmospheric air touched, it collected directly and slurred. Next, using this slurry, the horizontal magnetic field wet compaction was performed on impression magnetic-field-strength 795.8 kA/m (10kOe) and conditions with a compacting pressure of 7.8×10^7 Pa (0.8 t/cm²), and the abbreviation flat sector-like Plastic solid was acquired. Next, deoiling processing which heats in about 6.7 Pa (5×10^{-2} Torr) vacuum for 200 degree-Cx 2 hours was performed after feeding a Plastic solid into a vacuum heating furnace. The 1st step of vacuum heat-treatment of 600 degree-Cx 1 hour was succeedingly performed in the vacuum of about 6.7×10^{-6} to 4 Pa (5×10^{-6} Torr). Then, the 2nd step of vacuum heat-treatment of 1000 degree-Cx 1 hour was performed in the vacuum of about 6.7×10^{-6} to 4 Pa (5×10^{-6} Torr). Then, it cooled to the after [1070 degree-Cx 3 hour heating] room temperature in the vacuum of about 1.3×10^{-4} to 2 Pa (1×10^{-4} Torr), and the sintered compact was obtained. Next, after processing a predetermined configuration, in the argon ambient atmosphere, heat treatment of 900 degree-Cx 1 hour, and 550 degree-Cx 1 hour was performed once each, and it cooled to the room temperature. Next, barrel finishing was performed and plating pretreatment was performed after that. Then, electrolysis nickel plating (10 micrometers of average thickness) was covered, and the sintered magnet of this invention was obtained. The analysis value of said sintered compact is shown in Table 1, and magnetic properties and a consistency are shown in Table 2. Next, the flat sector-like sintered magnets 22 and 22 (thickness $t_m=4$ mm) of the produced pair

were respectively pasted up on the front face of York 23 and 25 made from a ferromagnetic after magnetization and through the magnetic opening 40 on the conditions with which magnetic properties are saturated, and the voice coil motor 20 of drawing 4 was produced. Next, in the magnetic opening 40 of a voice coil motor 20, when the opening flux density of the point P that are $(tg/2)$ and opening flux density shows peak value was measured, opening flux density useful as a voice coil motor was obtained.

(Example 3 of a comparison) The Plastic solid of the shape of an abbreviation flat sector produced in the example 3 was cooled to the after [1070 degree-Cx 3 hour heating] room temperature as it was in the vacuum of about 1.3×10^{-4} to 2×10^{-4} Pa (1×10^{-4} Torr), and the sintered compact was obtained. The flat sector-like sintered magnet (thickness $t_m = 4\text{mm}$) was produced like the example 3 henceforth. The sintered magnet of this example of a comparison was replaced with the flat sector-like sintered magnets 22 and 22 of an example 3, and was built into the voice coil motor 50 of drawing 4 . Next, in this voice coil motor, the opening flux density of the location where it is $(tg/2)$, and opening flux density shows peak value was measured. Consequently, compared with the voice coil motor of an example 3, the peak value of opening flux density was low 5%. The comparison of an example 3 and the example 3 of a comparison shows that it becomes the high density by which the amount of oxygen is reduced further and is equivalent to an $R_2\text{Fe}_{14}\text{B}$ mold intermetallic compound (the main phase) by performing the hypoxia process (wet compaction process) which can prevent advance of oxidation, and said vacuum heat-treatment, and the opening flux density of a voice coil motor can be improved further. Although the example 3 indicated the case where straight mineral oil was used, synthetic oil or vegetable oil may be used. Or the mixture of straight mineral oil, synthetic oil and synthetic oil, vegetable oil and straight mineral oil, synthetic oil, and vegetable oil may be used. calcium content can be made and 0.1% or less and a consistency can be made [the amount of oxygen of the R-T-B system sintered magnet which comes to blend R/D alloy powder 100%] into $7.57 - 7.70 \text{ Mg/m}^3$ (g/cm^3) for a carbon

content less than (0 is not included) 0.02% less than 0.2% by weight % by using together the wet compaction process of a publication, and said vacuum heat-treatment in the example 3. In 20 degrees C, iH_c of (BH) max of 302.5 - 398 kJ/m³ (38 - 50MGOe), and 1432.4 - 1989.5 kA/m (18-25kOe) and 95 - 98% of (H_k/iH_c) are realizable for coincidence.

[0016] (Example 4) After starting a tabular sample with a thickness of 1mm, respectively from each sintered magnet of examples 1-3 and the examples 1-3 of a comparison, it ground in thickness of 100 micrometers by wrapping processing. Then, it thin-film-ized in thickness of about 0.1 micrometers by Ar ion milling etc. Next, after carrying out the sequential set of said each thin film sample at transmission electron microscope FE-TEM (trade name: HF-2100) by Hitachi, Ltd., it gazed at the cross-section organization of each thin film sample on the acceleration voltage of 200kV, filament current 50microA, and conditions with a resolution of 19nm, and the cross-section photograph was taken. Moreover, identification of a presentation of the observed object visual field was performed on conditions with a beam diameter of 0.7nm using the EDX analysis apparatus (the product made from NORAN, trade name:VANTAGE). Drawing 1 is the photograph which photoed the cross-section organization which shows the typical main phase crystal grain part of the sintered magnet of an example 1 with said transmission electron microscope. Drawing 2 is a mimetic diagram corresponding to drawing 1. 1 and 21 were the main phase crystal grain of a core part from the electron diffraction result etc., and 5 and 25 are the surface sections which consist of two or more main phase crystal grain in the range whose diameter of average crystal grain is 0.5-50nm, respectively, and it turned out that 13 is a rare earth rich phase. The main phase crystal grain parts 10 and 30 exist through the rare earth rich phases 13 (point B etc.). Electron diffraction was performed in respect of [F] the point C of the surface section 5, Point D and Point E, the point A of a core part 1, and the core part 21, and the c-axis bearing difference between each measuring point was measured. A result is shown in Table 3. In Table 3, A-C means the c-axis bearing difference of Point A and Point

C. From Table 3, the include angle of c-axis bearing of a core part 1 (point A) and c-axis bearing of each **** crystal grain of the point C of the surface section 5, Point D, and Point E to make was 5-76 degrees. This result shows that the main phase crystal grain which constitutes the surface section 2 has random c-axis bearing. Moreover, the c-axis bearing difference of the core part 1 and core part 21 which exist through the rare earth rich phase 13 was less than 1 degree. This is most judged to be what shows the effectiveness in which the main phase crystal grain of the core part of the diameter of a large drop carried out orientation in the direction of an impression magnetic field each whole fines particle at the time of compression molding in a magnetic field.

[0017]

[Table 3]

	c 軸方位差 (度)
A - C	5
A - D	32
A - E	76
A - F	1

[0018] The cross-section organization of the sintered magnet of an example 1 gazed at microstructures other than drawing 1 and 2. The description of the microstructure of the sintered magnet of an example 1 is typically shown in drawing 3 . In drawing 3 , the main phase crystal grain parts 60, 70, and 80 exist through the rare earth rich phase 77. The main phase crystal grain parts 60 are drawing 1 and the same microstructure as 2. 61 is the main phase crystal grain of a core part, and the surface section which 65a becomes from the detailed main phase crystal grain of wrap plurality about a core part 61. The main phase crystal grain part 70 consists of main phase crystal grain 71 of a core part, and detailed main phase crystal grain 72 with which the surface section of a core part 71 is dotted. The main phase crystal grain part 80 consists only of one main phase crystal grain 85. Moreover, in the sintered magnet of an example 1, when the main phase crystal grain part was counted among one piece, 11 pieces and 83 main phase crystal grain partial 80 molds were observed [per / which was observed / 100 main phase crystal grain parts and the main phase crystal grain

part of 65 molds of drawing 3] for six pieces and the main phase crystal grain part of 70 molds of drawing 3 . Next, like the above, the cross-section organization of the sintered magnet of examples 2 and 3 and the examples 1-3 of a comparison was photoed, respectively, and was evaluated. In each cross-section photograph, when each **** crystal grain part was counted among one piece, the number ratio of the main phase crystal grain part which consists of two or more main phase crystal grain of per [which was observed] 100 main phase crystal grain parts, a core part, and the surface section was measured. A result is shown in Table 4. Table 4 shows that the number ratio of the main phase crystal grain part which consists of two or more main phase crystal grain of a core part and the surface section is less than 50% in the sintered magnet of examples 1-3.

[0019]

[Table 4]

	(芯部 + 表層部) / (主相結晶粒部分) の個数比率 (%)
実施例 1	24
実施例 2	17
実施例 3	7
比較例 1	55
比較例 2	58
比較例 3	51

[0020] Although the above-mentioned example indicated the voice coil motor, this invention is not limited to this but can constitute a useful spindle motor or a useful linear motor.

[0021] This invention is not limited to the R-T-B system sintered magnet manufactured only using R/D powder, but includes the R-T-B system sintered magnet which comes to blend R/D powder and ingot alloy powder by the predetermined ratio. In this case, in order to reduce a raw material cost, 10-100 weight section:90 - 0 weight section of : (R/D powder) (ingot alloy powder) is desirable, 30-100 weight section:70 - 0 weight section is more desirable, and especially 50-100 weight section:50 - 0 weight section is desirable.

[0022] Although Metal calcium was used for the reducing agent in the above-mentioned example, even when the hydride of calcium, the hydrides of Metals

Mg and Mg, or those mixture are used, while Mg content or (calcium+Mg) a content is made to less than (0 is not included) 0.02% of the weight, the sintered magnet which has the amount of oxygen, carbon content, and microstructure of the above-mentioned amount range of specification can be offered.

[0023] Although the above-mentioned example indicated the ring magnet which has a radial anisotropy, this invention is not limited to this. When the number of magnetic poles produces the R-T-B system sintered ring magnet which has the R-T-B system sintered ring magnet or path 2 polar anisotropy which has the surface multi-electrode anisotropy of four to 32 pole, notably high magnetic properties can be realized compared with the case where the alloy powder for R-T-B system sintered magnets by the conventional reduction/diffusion method is used.

[0024]

[Effect of the Invention] according to this invention as description, it was manufactured using the alloy powder for R-T-B system sintered magnets by the reduction diffusion method above -- it is cheap and the R-T-B system sintered magnet, ring magnet, and voice coil motor of high performance can be offered.

[Translation done.]

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is the photograph which photoed the typical cross-section organization of the sintered magnet of this invention with the transmission electron microscope.

[Drawing 2] It is a mimetic diagram explaining drawing 1 .

[Drawing 3] It is a mimetic diagram explaining the microstructure of the sintered magnet of this invention.

[Drawing 4] It is the A-A line view sectional view (b) of the important section sectional view (a) of the voice coil motor of this invention, and (a).

[Description of Notations]

1, 21, 61, 71, 85 A core part, 5, 25, 65, 72 Surface section, 10, 30, 60, 70, 80 13 The main phase crystal grain part, 77 Rare earth rich phase, 20 A magnetic pole boundary, 22 A flat-like magnet, 23 Upper York made from a ferromagnetic, 24 The stanchion made from a ferromagnetic, 25 Bottom York made from a ferromagnetic, 40 A magnetic opening, 26 A moving coil, 27 An arm, 30 A shaft, 31 The straight line, 32 which connect the core of a moving coil, and the core of a shaft The straight line, 50 which connected the core of a shaft, and the straight line along a magnetic pole boundary A voice coil motor, 65a The main phase microcrystal grain.

[Translation done.]

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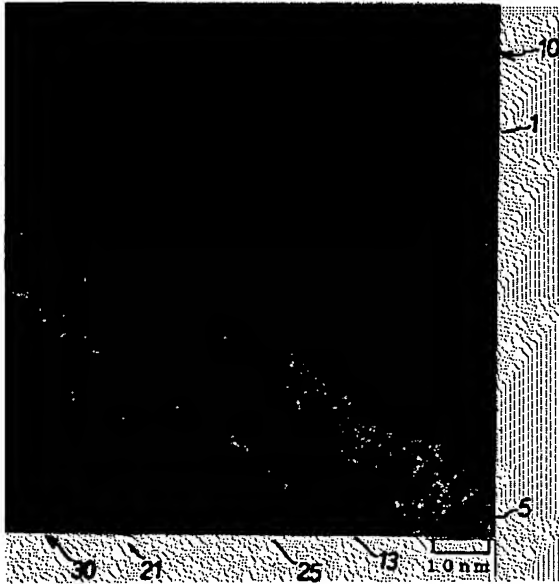
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DRAWINGS

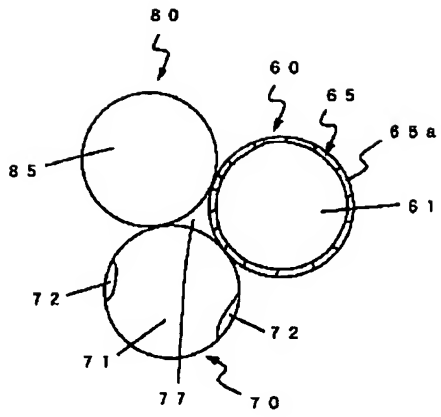
[Drawing 1]



[Drawing 2]

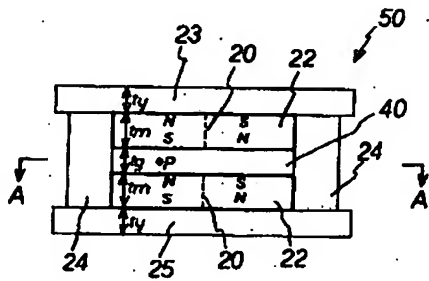


[Drawing 3]

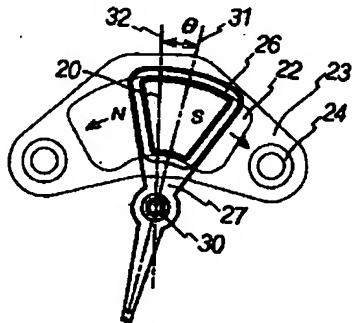


[Drawing 4]

(a)



(b)



[Translation done.]